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Cubic Silicon Carbide (3C SiC): Growth and Properties of Single Crystals and Polycrystalline Layers Prepared by Thermal Decomposition of Methyltrichlorosilane in Hydrogen

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This report results from a contract tasking Russian Academy of Sciences as follows: Investigate the process of growing cubic silicon carbide from the vapo phase in the form of single crystals and polycrystalline layers that can be used for the production of various semiconducting devices and passive elements.

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CUBIC Silicon Carbide (3C SiC): Growth and Properties of Single Crystals and

Polycrystalline Layers PREPARED BY THERMAL DECOMPOSITION OF

METHYLTRICHLOROSILANE IN HYDROGEN

by Stanislav N. GORIN and Ludmila M. IVANOVA

(service lifes of 10000 - 100000 hours) and working temperatures to 700 K;

thermal indicators for measuring maximal temperatures in a range of 360 to

1600 K in not easily accessible places at the objects of atomic industry and

space technology; high-temperature photodetectors for recor

ding power laser radiation; etc.

The high-purity polycrystalline layers of cubic silicon carbide may be used

as protective coatings on items of graphite or commercial (Acheson's) silicon

carbide as well as for making various structural elements such as chemically

resistant reactors, containers, mirrors, etc., employed in microelectronics

and power device-building.

This overview briefly describes the physico-chemical investigations into the

process of growing cubic silicon carbide (3C SiC) from the vapor phase by

thermal decomposition of methyltrichlorosilane in hydrogen. The method was

developed in the Baikov Institute of Metallurgy, Russian Academy of Sciences;

it ensures manufacturing single crystals and polycrystalline layers that can

be used for the production of various semiconducting devices and passive

elements.

2. BRIEF HISTORY

Silicon carbide was first obtained by Berzelius [1] as long ago as in 1824,

but the real birth year of SiC is 1892, when it was obtained and investigated

almost simultaneously by Moisson [2] and Acheson [3]. The latter, who

realized the technical importance of this hard and chemically stable product,

has developed a commercial technique [4] that up to now is the main method

for the production of silicon carbide. Tens of thousands of tonnes of SiC are

manufactured by this method annually, primarily for the

needs of abrasive, chemical, and electrotechnical industries, and metallurgy.

The Acheson process yields SiC crystals of the alpha modification. For sure

beta silicon carbide that had no marked conductivity at either room or higher temperatures.

Later (1947 - 1960), Kendall [9 - 11] used the same process in wider composition and temperature ranges. He conducted reactions between silicon tetrachloride and toluene in hydrogen and nitrogen atmospheres and in a partial vacuum; he used mixtures of silicon tetrachloride, benzene, and hydrogen; silicon tetrachloride and methane; silicon tetrachloride + methane + hydrogen; as well as methyltrichlorosilane (MTCS) and diethyldichlorosilane without hydrogen.

Kendall, like Moers, could not obtain crystals of more than 0.3 mm in size because the temperature on the crystal surface decreased with time and, with increasing current, the filament burnt out. All crystals were of cubic modification. However, the small size of crystals prevented direct measurement of electrical properties. The type of the charge carriers was determined from the sign of thermopower [10].

In 1955, Lely [12] reported on the development of a laboratory technique for the preparation of silicon carbide crystals by the sublimation of commercial SiC obtained by the Acheson process. Later [13, 14], Lely studied some properties of pure and doped crystals obtained by his method.

Lely's method permits one to obtain fairly large and perfect SiC crystals of hexagonal modification, but the yield is small and the apparatus is wieldy and power-consuming. For this reason, the attention of researchers was subsequently directed, from the one hand, toward the improvement of this method [15, 16, 24 - 28, 68, 69] and, on the other hand, toward searching out new, cheaper and more efficient, techniques for growing silicon carbide crystals, including 3C SiC. Studies in the development of the Van Arkel process continued [20, 21, 29 - 37]. In particular, attempts were undertaken to obtain SiC crystals inside a tube [31, 32, 35, 38 etc.] rather than on a filament; in the gas-suspension state [39]; in an electric arc

By the end of sixties, the attention of researchers was focused on the process of heteroepitaxy of 3C SiC on substrates such as Si, alpha silicon carbide, sapphire, etc., from various starting components (organosilicon compounds, silicon and carbon chlorides, hydrocarbons, etc.) [83 - 88].

In the seventies, the circle of researchers dealing with growing single crystals and polycrystalline films of 3C SiC became broader [75, 81, 89 - 95]; a large group of Japanese authors joined [96 - 98]. New data were accumulating; the properties of 3C SiC were actively studied. As starting components, they used methyltrichlorosilane [96], a mixture of silicon tetrachloride and propane [97]. It was found that 3C SiC films grow better on Si(100) substrates rather than on (111) or (110). Interesting results w ere obtained upon the deposition of SiC on 6H SiC, using methyltrichlorosilane and toluene [93]. It was found that, in this system, films of both alpha and beta modifications of SiC could be grown. Beta SiC is obtained if a silicon excess in the gas phase exists; the excess of carbon yields alpha SiC or an alpha and beta mixture.

In eighties, Japanese and American researchers suggested the use of a buffer (transition) layer during heteroepiaxial deposition of 3C SiC films on silicon substrates [99 - 102]. This technique was later widely applied in many works; it permits obtaining fairly thick and relatively perfect 3C SiC films (see, e.g., the proceedings of conferences on amorphous and crystalline silicon carbide that were held in 1987 - 1993 [103].

Because of a very small volume of the overview, we restrict ourselves to this very brief enumeration of the methods that permit producing cubic silicon carbide crystals and consider in more detail the method that was developed in the Baikov Institute of Metallurgy. The principal results of the investigations performed in the Baikov Institute were published in [104 - 126].

Reactions (1) - (3) do not rule out the possibility of occurring other, side

reactions. We studied [112] the composition of the solid, liquid, and gaseous

products that formed upon the decomposition of MTCS in hydrogen at T = 1523 -

1873 K. The reaction was found to occur with a high yield (60 - 80%) and be

accompanied by the formation of the appropriate amount of HCl according to

(1). At the same time, reactions leading to the formation of some simple

chlorosilanes and hydrocarbons occur.

Chromatography and chemical analysis show that the main liquid products of

MTCS decomposition are $SiHCl_3$ and $SiCl_4$. Elevated temperatures favor more

complete transformation of MTCS into SiC; decreasing temperature leads to

side reactions that result in the formation of liquid products. In gaseous

products of the reaction, small amounts of methane (1.18%), ethane and

ethylene (0.31%), and acetylene (0.35%) can be found.

The presence of these compounds can be explained by the occurrence of

intermediate and side reactions. For example, upon the interaction of MTCS

with hydrogen, trichlorosilane and methane can form by reaction

(4) $CH_3SiCl_3 + H_2 \iff CH_4 + SiHCl_3$.
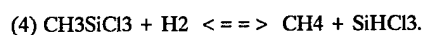
The interaction of MTCS with HCl that evolves by reaction (1), silicon

tetrachloride and methane can form by reaction

(5) $CH_3SiCl_3 + HCl \iff CH_4 + SiCl_4$.
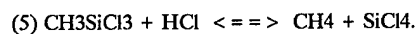
The calculation of the changes in the Gibbs free energies for reactions (4)

and (5) shows that the reactions can occur in a wide temperature range (300 -

1000 K). The probability of their occurrence decreases with temperature, but

they are still possible at above 1000 K, for the Gibbs free energy still

remains negative.

We see, thus, that although hydrogen does not participate in the total

(3) the diffusion range (above 1873 K). The activation energy decreases to

about 12 ccal/mol at T = 1873 K.

Thus, in the range of relatively low temperatures (up to 1723 K), where

polycrystalline layers of 3C SiC form, the rate of crystallization is

reaction-controlled; at higher temperatures (above 1873 K), where separate

crystals form, the limiting stage of SiC growth is diffusion, which controls

the rate of the supply of the reacting agents to the surface of growing

crystals and the rate of the removal of the reaction products.

4. LABORATORY APPARATUS FOR GROWING CUBIC SILICON CARBIDE SINGLE CRYSTALS AND POLYCRYSTALLINE FILMS

The laboratory apparatus used for growing pure and doped 3C SiC by thermal

decomposition of methyltrichlorosilane (MTCS) consists of an all-glass, air-

and vacuum-tight system fabricated primarily from hard borosilicate glass,

quartz, and Teflon. We avoided using metallic parts because MTCS is a very

reactive substance. The dismountable parts of the apparatus, such as the

vessels for MTCS and liquid doping compounds (evaporators), are connected to

the setup through ground-glass joints or rubber O-rings ensu

ring the air-tightness of the apparatus. The system permits the introduction

of flow-metered streams of hydrogen, saturated with the vapors of MTCS and

doping compounds, into the reaction chamber. All flowmeters are calibrated in

situ. The apparatus has a modular construction, which ensures rapidly testing

and disassembling the setup for cleaning. Each of the moduli can be evacuated

and washed by hydrogen independently of the other parts.

The high temperatures developed in the reaction zone (1700 - 1900oC) and the

presence of gaseous hydrogen chloride that forms by reaction (1) impose

severe restrictions on the chemical stability and purity of the parts located

in the reaction chamber. Quartz, graphite, and Teflon serve as the main

merged into Dewar vessels with liquid nitrogen; this permits one to

simultaneously remove all condensable and adsorbable gaseous impurities.

The method is based on the high absorption capacity of silica gel and

activated carbon at low temperatures; thus, it is known that at -195oC, one

gram of activated carbon can absorb 155 cm3 of nitrogen, 230 cm3 of oxygen,

133 cm3 of hydrogen. The content of oxygen and nitrogen in hydrogen purified

by this method is known to be less than 10-8 vol.%.

When purifying hydrogen used for growing silicon carbide, special attention

should be given to the removal of nitrogen because this impurity is the main

shallow donor impurity in SiC. According to literature data, even the best of

activated carbons adsorbs nitrogen less effectively than silica gel. For this

reason, we used a system of three sequentially connected columns to purify

hydrogen. The first column is filled with silica gel and is used for

preliminarily drying and purifying commercial hydrogen supp

lied from a baloon. The second column is filled with activated carbon; we

cannot manage without this column because silica gel adsorbs oxygen much

worse than activated carbon does, and we know that oxygen, like nitrogen, is

electrically active in 3C SiC [123]. The third column is again filled with

silica gel; this is intended for improving the purification of hydrogen from

nitrogen and also serves as a mechanical filter for trapping carbon dust that

can be carried-over with hydrogen from the column with act

ivated carbon.

Before the silica gel and activated carbon are placed into the columns, they

must be carefully cleaned in chemically pure or high pure hydrochloric acid

(1 : 1), then must be washed in doubly distilled water until any traces of

chlorine ions vanish, and dried first in a hydrogen flow and finally in a

vacuum. The columns must be "trained" (evacuated to a residual vacuum of an

order of 10-2 mmHg) for 0.5 - 1.0 h prior to each run without heating and

after each 5 - 10 runs, with heating to 90 - 120oC for 3 to

4 h

chamber through a water-cooled ground-glass joint (to facilitate cleaning

it).

The as-assembled (soldered) apparatus is evacuated with a roughing-down pump

until the apparent leakage (i.e., the fall of vacuum after pumping is

stopped, caused by the presence of water vapors and other volatile impurities

in the setup) disappears. The adequately purified setup is capable of keeping

vacuum not worse than 0.1 mmHg for a few weeks if the initial vacuum was 0.01

- 0.001 mmHg.

The growth process may be initiated after the starting components

(methyltrichlorosilane and the doping compounds if necessary) are put into

the evaporators and the whole apparatus is washed with purified hydrogen. The

process consists of passing a vapor-gas mixture of a specified composition at

a constant flow rate through the reaction chamber past the filament heated to

a specified temperature.

The growth of crystals in a flow system is controlled by many parameters such

as the filament temperature; reaction mixture composition; flow rate of the

gaseous mixture (and the linear velocity of its motion in the chamber) ;

temperature gradient; the shape of the heated surface; conditions for crystal

nucleation; and some other factors such as the effects of impurities, thermal

and concentration shocks, etc.

In order to determine the optimal conditions for growth single crystals and

polycrystalline films, large systematic work was done.

Using temperatures below 1650^oC results in the deposition of numerous small

crystals grown together or, at temperatures $1250 - 1450^oC$, of a continuous

dense polycrystalline film. At $1650 - 1750^oC$, separate euhedral crystals

grow, which, frequently, have the form of regular hexagonal well-faceted

platelets 3 - 5 mm in length and 1 - 1.5 mm in thickness.

After the growth run is stopped, the hydrogen flow rate is increased to 200 -

300 liter/h, the filament is switched-off, and the system is blowed out with

hydrogen for at least 0.5 h in order to carefully remove the undecomposed

MTCS and the products of its decomposition.

5. GROWTH MORPHOLOGY OF BETA SILICON CARBIDE SINGLE CRYSTALS PRODUCED BY THERMAL DECOMPOSITION OF METHYLTRICHLOROSILANE

At temperatures of about 1650 - 1750oC, the deposit consists, as was

mentioned above, of separate faceted crystals. In the general case, crystals

have various shapes (which frequently does not exhibit visible symmetry) and

various degrees of perfection, which depends both on the growth conditions

and the type of the crystal. However, it was found that the majority of

crystals obtained under any conditions could be classified to three main

types, which were called "platelets", "prisms", and "skeletal crystal

s". Crystals of the first type are flattened in one of the <111> directions

and frequently have a characteristic form of regular hexagonal platelets with

one most developed mirror face. Crystals of the second type are extended

along a direction <110> and have from 6 to 10 faces of this crystallographic

zone (pseudoprism faces). Crystals of the third type have a few reentrant

dihedral angles that give the crystal a characteristic skeletal appearance.

The facets were identified by X-ray diffraction, etching , and goniometry

(performed on a Goldschmidt two-circle optical goniometer with an accuracy of

1 min of arc). An investigation of the morphology and sectorial structure

(i.e., the structure of growth cones or pyramids caused by impurity

segregation) of pure and doped crystals of 3C SiC in various sections showed

that

(1) the growth direction of the majority of crystals is <110>;

(2) the following faces are formed on most crstals: C{111} (positive

strong birefringence when being inspected in polarized light. It was shown

that even most perfect crystals contain a characteristic structure (th

at was called twin structure) in the form of a few MTL passing through the

crystal vertex looking in the growth direction.

Skeletal crystals is a special case: they contain, in addition to coherent

twin boundaries on {111} planes, twin boundaries of higher order (symmetric

third order tilt boundaries of the (110)[110] type with a tilt angle of

31o35', twin plane {552}). These boundaries appear as a result of multiple

twinning on nonparallel {111} planes. From four possible types of the mutual

arrangement, only one type occurs in all crystals without exception. Etching

and goniometry indicate that the boundaries are of the type

Si{552} - Si{552}, i.e., they contain dislocations with "wrong" bonds of the

A - A type (alpha dislocations). This is a direct consequence of the

crystallographic polarity of the sphalerite structure. Another related effect

of the polarity is the absence of multiple twins with the apparent fivefold

symmetry that are quite typical for diamonds. Multiple twins in 3C SiC always

involve the above-described {552}-type boundaries.

A comparison of the faces appearing on real crystals with those

characteristic of the ideal form shows that MTL can locally increase growth

rate of 3C SiC crystals. Measurements of real growth rates of various faces

by studying sectorial structure of crystals revealed that

(i) MTLs have their own growth rates VMTL; when an MTL intersects a face

whose growth rate is smaller than Vo = VMTLcos a, where a is the smaller

angle between the MTL and the face, the face transforms; it is replaced by

two induced vicinal faces whose growth rates are related to Vo as V = Vocos

b, where b is the angle between the induced and vicinal faces. The site where

the MTL emerges onto the surface of the face coincides with the edge between

the induced faces (which form a ridge). For the Si{111} fac

es, whose growth rate is about VMTL/3, the tilt of the vicinal faces

{1 1 1 + e} relative to Si{111} is 0.5 - 1.0o. For the C{111} faces, whose

faces. Microtwin lamellae disclose this shell, and the habit and faceting of
all types of 3C SiC crystals are unumbiguously controlled by their twin
structure, i.e., by the thickness of the MTLs present and their type (i.e.,
by whether the number of twin planes is even or odd).

6. EFFECTS OF CRYSTALLOGRAPHIC POLARITY: IMPURITY SEGREGATION AND ETCHING

Pure beta silicon carbide single crystals are transparent and amber yellow.
Some impurities make them dark (black or green). This permits one to easily
follow their behavior in crystals. It was found by visual inspection that Al,
B, and N are distributed in crystals inhomogeneously: the crystals have
"sectorial" structure caused by preferential absorption of different
impurities by different faces. The study of the sectorial distribution of
impurities was performed in sections cut along growth pyramids of c
rystal faces (which are also called growth loci or growth cones) prepared in
such a way that the section pass through the initial point of growth (the
"root" of the crystal by which the crystal is attached to the graphite
filament). The distribution of nitrogen and boron was studied in detail.
Because both impurities are electrically active in SiC, their distribution
found visually was confirmed by electrical measurements.

It was found that nitrogen is "absorbed" preferentially by C{hhl} and C{hkk}
faces and much weaker, by Si{111}, Si{211}, and {100} faces, whereas boron is
absorbed most actively by Si{111} faces, somewhat weaker by Si{211} faces,
and much weaker, by C{hhl}, C{hkk}, and {100} faces, i.e., preferential
absorption of nitrogen is observed in growth pyramids of "carbon" faces
(positive hkl forms) and it is absent in "silicon" faces (negative hkl
forms); for boron, the picture is opposite: this impurity is prefer
entially absorbed by negative hkl forms. Cube faces {100}, which are nonpolar
in the sphalerite structure, exhibit no preferential absorption for either
nitrogen or boron. Aluminum is distributed similar to boron

triangular etching figures at the faces of negative hkl forms (carbon faces),

whereas such figures are absent at the faces of positive hkl forms (silicon

faces); moreover, the etching figures are depressions on silicon faces and

they are hills on carbon faces. Etching pits on cube faces are

lens-shaped, with lens axes aligned in only one of the two possible <110>

directions; etching pits at the opposite {100} faces are mutually

perpendicular. In some etchants (PbO. chlorine), the etching rates of carbon

faces are many times greater than those of silicon faces. Upon oxidation in

wet oxygen (at 1200oC), the oxide film on carbon faces is approximately twice

as thick as that on silicon faces.

7. POLYCRYSTALLINE LAYERS OF CUBIC SILICON CARBIDE: PREPARATION AND PROPERTIES

The yield, deposition rate, and structure of polycrystalline layers of 3C

SiC (PCL) that are obtained by reaction (1) at low temperatures depend on a

number of parameters, the most important of which are (a) the temperature at

which the deposition is performed (T, oC); (b) the concentration of the

starting component in the gas phase (C, g/liter); (c) the flow rate of

hydrogen in the reaction chamber (V, liter/h); and the area of the deposition

surface (S, cm2). When studying the growth of PCL in our work,

these parameters were varied in the following limits: the deposition

temperature, 1250 - 1600oC; the MTCS concentration in hydrogen, 0.2 - 1.7

g/liter; and the gas flow rate, 3 - 30 liter/h.

Our investigations [105, 113] indicate the following conditions to be optimum

for the production of pure dense polycrystalline layers of 3C SiC: the

deposition temperature, 1400 - 1500oC; the concentration of MTCS in hydrogen,

0.5 - 1.5 g/liter; and the flow rate of the gaseous mixture, 6 - 10 liter/h.

Under these conditions, the 3C SiC PCL have a smooth surface and fine

microstructure: the yield of SiC varies from 65 to 70% The duration of the

reflectance in the range of 10 to 14 microns: R = 90 %.

Measurements of the electrical properties show that pure 3C SiC PCLs have electronic conductivity; their resistivity varies from 0.1 to 1000 Ohm cm; the charge carrier concentration is n = 1.4 x 1016 - 5 x 1017 cm-3.

Pure polycrystalline beta silicon carbide has high thermal conductivity. The maximum value is 5 W/(K cm) at 110 K; with increasing temperature, thermal conductivity decreases, but still remains sufficiently high: 2.5 W/(K cm) at room temperature.

The thermopower of pure polycrystalline beta silicon carbide with a charge carrier concentration of n = 5 x 1017 - 1.8 x 1016 cm-3 ranges from -500 to -600 microvolt/K.

8. DOPING POLYCRYSTALLINE CUBIC SILICON CARBIDE

Doping polycrystalline beta silicon carbide with nitrogen and boron was performed directly during growth by thermal decomposition of methyltrichlorosilane.

The most suitable compounds for doping with nitrogen are nitriles, which are the derivatives of aliphatic and aromatic acids. They only contain carbon, hydrogen, and nitrogen, i.e., there is no danger of contamination by foreign impurities when using nitriles. The nitriles are stable compounds that can be purified by distillation or rectification in air under an atmospheric pressure.

From the variety of nitriles available, we selected acetonitrile as the simplest one [114, 118]. In order to introduce it into the reaction chamber, a separate evaporator was used, which permitted us to easily vary its concentration in the gas phase.

(c) all samples grown at boron concentrations lower than 1.5 mg/liter

retained electronic conductivity. Distinct inversion of the conductivity

type from n to p type occurs at a boron concentration of 2.5 mg/liter. The

samples had in this case a resistivity of 2600 Ohm cm. According to

activation analysis, the maximum content of boron in beta SiC is about 4 x

10^{19} cm-3;

(d) the introduction of excess silicon (in the form of silicon tetrachloride

or trichlorosilane) into the gas phase strongly affects the process of doping

beta SiC by both donor and acceptor impurities, decreasing the degree of

doping by an order of magnitude or even greater.

Doping with boron substantially improves photoelectric properties of

polycrystalline samples of 3C SiC [125]: it increases the impurity

conductivity in comparison with that of pure beta SiC; it increases the

exponent in the expression for the lux-ampere characteristics for both the

intrinsic and extrinsic conductivities; the temperature dependence of the

photocurrent becomes activative (the photoresponse increases with increasing

temperature); the activation energy increases with increasing concentration

of boron. The photoresponse times are much shorter in these samples (of

about few seconds).

The photoelectric properties of polycrystalline cubic silicon carbide may be

of interest for the production of photodetectors that can operate under

extreme conditions such as high temperatures, high pumping levels, reactive

media, severe irradiation, etc.

9. SOME PARAMETERS AND APPLICATIONS OF PURE 3C SiC SINGLE CRYSTALS

Single crystals of pure cubic silicon carbide prepared by thermal

decomposition of methyltrichlorosilane in hydrogen have the following

= working temperature: -60 to +80oC.

(2) High-temperature photodetector.

It can be used for detecting high-power laser radiation; operating at

elevated tempeartures; precision detecting of short-wavelength light signals

(up to ultraviolet range). Its main parameters are as follows:

= spectral range: 280 - 650 nm;

= spectral maximum: 330 - 480 nm;

= working temperature: 20 - 350oC;

= photosensitivity: 10 mA W-1 in valve regime; 1000 mA W-1   in

photoinjection regime; 50 mA W-1 in photodiode regime;

= response speed: to 5 x 10-7 s;

= service life: > 105 h;

= maximal irradiation power: 40 W (stationary);

                100 W (pulsed);

=

(3) Gamma detector.

A high-temperature detector of high-power radiation flows; intended for

detecting gamma radiation (> 108 rad) in temperature range from -200 to

+400oC. It has a highly linear dynamic characteristic; no degradation under

high irradiation doses.

(4) Violet emitter.

May be used as a standard light source for calibration and stabilization of

photometric tracts in opto-electronic systems.

CONCLUSIONS

IP0,0,1016,1016;SC0,600,0,600;

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